PHOSPHORUS FERTILIZATION OF RYEGRASS WITH TEN PRECISELY PREPARED MANURE BIOCHARS

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ABSTRACT. *Biochars made from livestock manures need to be better understood in relation to their high nutrient contents. Accordingly, the objective of this investigation was to assess the plant availability and agronomic efficacy of phosphorus (P) contained within chemical fertilization compared to the P contained in ten precisely prepared and characterized manure-derived biochars. Two identical greenhouse experiments were conducted using ryegrass (*Lolium multiflorum *Lam.). The ten biochars were created from dairy, swine, beef, turkey, and chicken manures. Each manure feedstock was converted into biochar at two temperatures (350°C and 700°C). The soil was a sand (Uchee series; loamy, kaolinitic, thermic Arenic Kanhapludults) with a low P content. The biochar-amended treatments received P on a basis of 50 mg P_2O_5 kg^{-1} soil. The biochar treatments were compared to chemically fertilized treatments of 0 to 150 mg P kg^{-1} soil. The biochars did not contain yield-limiting levels of EC, pH, Zn, or Cu. Phosphorus in the manure-derived biochars was readily plant available. The biochars generally produced ryegrass yields similar to chemical fertilizer. The chicken 350°C biochar produced the highest ryegrass yield, and the swine 350°C biochar produced the lowest ryegrass yield. For the biochar treatments, the ryegrass Cu and Zn concentrations were within normal ranges, and they were not significantly different from the simple chemical fertilization treatments. Thus, for ryegrass growth, manure biochars can be used for their P-supplying capacities. This can be important to manure management, crop fertilization, and conservation of global P resources.*

Keywords. *Manure management, Mehlich 3 extraction, Plant nutrients, Pyrolysis, Thermochemical conversion.*

Manure management for crop application has long been vexed by the problems associated with transporting its high bulk weight relative to its nutrient content, the presence of pathogenic microorganisms, and phosphorus (P) to nitrogen (N) (P/N) ratios higher than needed by the fertilized crop. One potential way of dealing with these issues is to pyrolyze livestock manures into biochar. Converting livestock manures into biochar via pyrolysis would greatly increase its density and eliminate its pathogen content. Moreover, biochar could be readily blended with supplemental N to obtain the desired ratios of N and P. This option for manure management can now be visualized in the broader context of soil management where biochar is no longer a novel amendment. Non-manure based biochar has moved from a relatively obscure aspect of soil science to one of great international interest (Glaser et al., 2002; Lehmann, 2007; Marris, 2006; Novak et al., 2012).

Biochars have been generally discussed and tested based on their carbon (C) content. Typical feedstocks have included wood and crop residues. To a much less extent, there has been interest in the possibility of using livestock manures as a biochar feedstock (Cantrell et al., 2007). In contrast to many biochars used for the important functions of soil quality and C sequestration, livestock manures are very nutrient dense, especially P (Cantrell et al., 2012; Hass et al., 2012). These high nutrient contents cause reason for both exuberance and caution. For example, the high level of P associated with livestock manures has caused substantial problems in some areas, and it has required P application and accumulation to be an important aspect of farm nutrient management plans (Revell et al., 2012). Additionally, high levels of copper (Cu) and zinc (Zn) are often present in manures (Cantrell et al., 2012). Yet, the high density of P and the lack of pathogens are very attractive attributes for a good P fertilizer material. Although the actual nutrient concentrations of manure biochars vary considerably, the content of P relative to N is nearly always high compared to crop actual N and P needs (Rajkovich et al., 2012). As such, the plant availability of P in livestock manure biochars is an important issue. If it is plant available, manure biochar could be considered as an alternative material for providing P to growing crops. The recent and limited research for specific manure biochars, soils, and crops suggests that the P in biochar is plant available (Revell et al., 2012; Wang et al., 2012). Accordingly, manure biochars would have to be managed as fertilizers rather than as a carbon-building amendment. However, nearly all of the reported biochar studies have added biochar at levels that would far exceed the manageable P fertilization. Moreover,

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feedstocks and pyrolysis conditions of the reported manures biochars vary greatly.

No study has reported the plant fertilizing effectiveness of "precisely prepared" biochars from the five major livestock manures (beef, dairy, chicken, turkey, and swine). The objectives of this investigation were to assess: (1) the plant availability and agronomic efficacy of P contained in ten precisely prepared and characterized manure-derived biochars, and (2) the resulting accumulations of Cu or Zn in both the soil and plant when fertilized with these biochars.

Materials and Methods

The efficacy of manure-based biochar was assessed using annual ryegrass (*Lolium multiflorum* Lam.) grown on a low P soil of the eastern Coastal Plain of the U.S. This greenhouse study, conducted in Florence, South Carolina, was a repeated measures experiment: the initial experiment began in January 2011, and the second experiment was started two weeks later. Each experiment was arrayed in a randomized complete block design. There were ten biochar treatments and three P-level chemical treatments; additionally, there were both N-only and control (soil-only) treatments. For each experiment, there were four replications; these in total constituted 60 experimental units.

The soil for the experiment was from the top 15 cm depth of a sandy soil (Uchee series; loamy, kaolinitic, thermic Arenic Kanhapludults). The soil was collected from a forest site that was recently harvested for timber. As would be anticipated, the pH was low (4.5). Thus, before the biochars were added, the soil was limed to a target pH of 6.0 by mixing and equilibrating with 1.99 g lime kg^{-1} soil. The soil and any amendments were placed into plant growth pots. The pots were 7.6 L volume; their diameter was 20 cm, and their height was 24 cm. Each pot received 6.1 kg of soil. The soil of each biochar treatment pot received biochar at a rate equivalent to 50 mg P_2O_5 kg^{-1} soil. The applied biochar amounts were determined based on previous knowledge of the biochars' P concentration (Cantrell et al., 2012). Non-biochar treatments received fertilizer treatments that included three P levels, N only, and a blank (table 1). The P treatments were 0, 50, and 150 mg P_2O_5 kg^{-1} soil; this is equivalent to 22.2 and 66.6 mg actual P kg^{-1} soil for the 50 and 150 mg P_2O_5 fertilizer treatments, respectively. The P was added as calcium hydrogen phosphate dehydrate ($CaHPO_4{\cdot}2H_2O$). The chemical fertilizer treatments also received 50 mg K_2O as potassium chloride. Nitrogen was added at a rate of 50 mg N kg^{-1} soil to all biochar and fertilizer treatments. The N was added as ammonium chloride (NH_4Cl). The blank treatment was unamended soil. The final pH of the blank treatment was 5.81, and the final pH of the P-0 treatment was 5.33. The beef 700°C biochar amended soil had a pH of 6.4; the other biochar amended soils had pH values ranging from 5.34 to 5.60.

Table 1. Fertilizer treatments for the ryegrass greenhouse biochar experiment.

Fertilizer Treatment	P_2O_5 (mg kg^{-1})	K_2O (mg kg^{-1})	NH_4Cl (mg kg^{-1})
P-0	0	50	50
P-50	50	50	50
P-150	150	50	50
Blank	0	0	0
N only	0	0	50

The five manure biochar types were created from beef (paved feedlot), dairy, swine, turkey, and poultry manure feedstocks. Each of the manures was converted into biochar by slow pyrolysis at two temperatures, either 350°C or 700°C. The farm origins of each manure, the pyrolysis process, and the specific characteristics of the biochars were described in detail by Cantrell et al. (2012). These ten biochars were produced using a well defined protocol (Cantrell et al., 2012; Cantrell and Martin, 2012a, 2012b). Briefly, the feedstocks were pyrolyzed in a nitrogen atmosphere for 120 min equilibrium held at either 350°C or 700°C. After charring, the samples remained in an inert atmosphere but were allowed to cool to room temperature for subsequent removal from the retort. The biochars were then homogeneously subsampled for analysis. Biochar samples (homogenized from three or four retort runs, depending on feedstock type) were analyzed previously for pH, EC, total P, total K, and ash content (Cantrell et al., 2012). Briefly, pH and EC were measured in a 1% (w v^{-1}) suspension in deionized water prepared by shaking at 100 rpm for 2 h, the ash content of each biochar was measured as the residue remaining after combustion at 600°C, and total nutrients were based on wet acid digestion (conc. HNO_3 + 30% H_2O_2). Biochar samples also were measured for their Mehlich 3 (M3) extractable P and K. Following a modified procedure of Mehlich (1984), 0.5 g of biochar was added to 25 mL of M3 extraction solution, shaken for 10 min, and then allowed to settle for 2 h prior to filtering with a 0.45 µL syringe filter. The M3 extractions were single point measurements of the mixed biochar sample (three retort runs).

Each plant growth pot was planted to gulf annual ryegrass with 1.6 g of seed. This constituted a rate of 48.8 kg seed ha^{-1}. The pots were irrigated twice a day using an automated drip irrigation system for a daily pot total of 0.4 cm water. Plant height measurements were taken weekly. Ryegrass was harvested three times in experiment 1 and four times in experiment 2. Ryegrass was harvested by hand using scissors; it was cut to a height of 2.5 to 3.5 cm above the soil surface. In experiment 1, the harvest days were 24, 38, and 51 days after planting; similarly in experiment 2, the harvest days were 21, 35, 44, and 51 days after planting. For each harvest, plant dry weights were recorded after they were dried in an oven at 60°C for three days. The dried samples were ground before analysis using a cyclone mill. Ryegrass harvests were composited at the end of the experiment for analysis. Plant samples were analyzed for both C and N using a Leco TruSpec CN analyzer (Leco Corp., St. Joseph, Mich.). For other nutrient plant analyses, dry samples were digested with nitric acid and hydrogen peroxide using an Auto-Block (Peters et al., 2006). Subsequent to the digestion, total elemental analyses were accomplished with an inductively coupled plasma atomic emission spectrometer (ICP-AES; VistaPro, Varian, Inc., Walnut Creek, Cal.). These analyses included P, potassium (K), calcium (Ca), magnesium (Mg), Zn, and Cu. To assess one potential impact of biochar applications to downstream utilization of ryegrass (e.g., combustion), a portion of the dried and milled ryegrass was analyzed for energy density or higher heating value (HHV) using a Leco AC500 Isoperibol Calorimeter (Leco Corp., St. Joseph, Mich.) following ASTM Standard D5865 (ASTM, 2006). The ryegrass biomass collected from the soil-only treatment was not analyzed for HHV due to sample availability.

After the final plant harvest, soil cores were collected from the surface to the bottom of each pot. Samples were combined and mixed. The soil samples were air-dried for one week, ground, and sieved using a 2 mm sieve. Soil samples were analyzed for C and N on the Leco CN analyzer. After a Mehlich 3 extraction of the soils, the extract solution was analyzed using the ICP-AES to determine the soil content of P, K, Ca, Mg, Zn, and Cu (Mehlich, 1984).

The data were statistically analyzed using SAS version 9.2 (SAS, 2002). Analysis of variance was done using the GLIM-MIX procedure. The replications were considered random; treatments were considered fixed. Treatments were analyzed using the least squares mean (LSM) method. In the ANOVA, feedstocks were highly significantly different, but temperatures were not significantly different. While the treatment interactions were only weakly significant, presentation of their means seemed important. The treatment differences of analyzed variables were compared using the pdiff option. The T value grouping for treatment LSM was at $p \leq 0.05$.

Results and Discussion

Characteristics of the Biochars

The P content of the biochar was dramatically higher than the values associated with woody or crop residue based biochars, such as used by Novak et al. (2012) (table 2). Even within the manure feedstocks, the biochars varied in P contents five-fold. The mean was 26.9 ±15.0 g P kg^{-1} biochar. The lowest concentration, 10.0 g P kg^{-1} biochar, was in the dairy biochar produced at 350°C. The highest P concentration, 59.0 g P kg^{-1} biochar, was in swine biochar produced at 700°C. The rank of total P was according to the type of livestock digestive system. The bovine with ruminant digestion exhibited the lowest total P concentrations, the swine with monogastric digestion generated the greatest total P concentrations, and the poultry species were intermediate.

With regard to extractable P, the ranking according to type of livestock digestive system was similar to the total P (table 2). The biochars had an average Mehlich 3 extractable P of 1.58 ±0.79 g P kg^{-1}. Similar to the total P, the Mehlich 3 P varied by six-fold, with the lowest content of 0.46 g P kg^{-1} reported for dairy 700°C (rather than 350°C). The highest content of Mehlich 3 extractable P, 3.02 g P kg^{-1}, was found in the swine 700°C biochar. In contrast to the quantity of total P, the percentage of Mehlich 3 extractable P was very different. While the lowest extractable percentage was 2.72% for dairy 700°C, dairy 350°C had the highest extractable P (11.20%). Coincidentally, these values represent the overall range for the percentage of Mehlich 3 extractable P.

While there were considerable variations in the literature reported values, which were derived from biochar experiments from different continents and livestock management methods, the P contents for the biochars used in this experiment are somewhat similar to the literature reported values. Wang et al. (2012) reported the P content of dairy manure biochar pyrolyzed at 350°C to be 6.16 g kg^{-1}. The dairy biochar made at 300°C by Rajkovich et al. (2012) had a P content of 5.4 g kg^{-1}. However, their P concentration increased to 8.31 g kg^{-1} when the pyrolysis temperature increased to 500°C. This concentration was strikingly close to the 8.14 g kg^{-1} reported by Uzoma et al. (2011) for a Japanese dairy biochar produced at 500°C. These reported P values are similar to but somewhat lower than the 10.0 to 16.9 g P kg^{-1} for the dairy biochar in the current investigation. For poultry manure biochar, much higher values were reported. Revell et al. (2012) measured a P concentration of 43 g kg^{-1}; their biochar was made at 450°C via fast pyrolysis. Rajkovich et al. (2012) reported biochar made from poultry manure at 300°C to 600°C to have total P concentrations of 18 to 31 g kg^{-1}. Thus, these values are very similar to the values of 20.8 to 31.2 g P kg^{-1} for the poultry biochar in the current investigation. Trumping these concentrations were those associated with swine biochars. The swine manure P concentrations were distinctly higher, ranging from 38.9 to 59.0 g kg^{-1}, yet they were slightly lower than the P concentration of the biochar produced by Tsai et al. (2012) at temperatures of 400°C to 800°C, which ranged from 61 to 77 g kg^{-1}.

The biochar concentrations of the primary plant nutrients N and K, the ash content, and the EC and pH values are presented in table 2. For N, the biochars had a mean of 28.1 ±10.4 g N kg^{-1} biochar. The N concentrations of the biochar

Table 2. Phosphorus (P), potassium (K), and nitrogen (N) contents and other chemical characteristics of the ten manure biochars.

Feedstock	Pyrolysis Temperature	Total P[a] (g kg^{-1})	M3 P[b] (g kg^{-1})	M3P:TP (%)	Total K[a] (g kg^{-1})	M3 K[b] (g kg^{-1})	N (g kg^{-1})	Ash[a] (%)	pH[a]	EC[a] (µS cm^{-1})
Dairy	350°C	10.0	1.12	11.20	14.3	2.59	26.0	24.2	9.2	538
	700°C	16.9	0.46	2.72	23.1	3.00	15.1	39.5	9.9	702
Beef	350°C	11.4	1.07	9.39	32.0	4.65	36.4	28.7	9.1	713
	700°C	17.6	0.60	3.41	49.1	5.97	17.0	44.0	10.3	1140
Chicken	350°C	20.8	1.81	8.70	48.5	6.59	44.5	30.7	8.7	1405
	700°C	31.2	1.89	6.06	74.0	8.85	20.7	46.2	10.3	2217
Turkey	350°C	26.2	1.68	6.41	40.1	4.91	40.7	34.8	8.0	651
	700°C	36.6	1.83	5.00	55.9	6.10	19.4	49.9	9.9	981
Swine	350°C	38.9	2.36	6.07	17.8	2.41	35.4	32.5	8.4	216
	700°C	59.0	3.02	5.12	25.7	3.29	26.1	52.9	9.5	194

[a] Portion of data previously reported by Cantrell et al. (2012).
[b] Mehlich 3 extractable nutrients.

ranged from 15 to 45 g kg^{-1} biochar. However, compared to the 700°C biochars with a mean concentration of 19.7 ±4.2 g N kg^{-1}, the 350°C biochars were higher in N content, with a reported mean of 36.6 ±6.95 g N kg^{-1} biochar. For total K, the concentrations ranged from 14.3 to 74.0 g kg^{-1} biochar. The mean was 38.1 ±19 g K kg^{-1} biochar. The K content was a significant factor in the biochar EC (Cantrell et al., 2012). The EC varied by more than ten-fold: the lowest value was 194 µS cm^{-1} measured in the swine biochar produced at 700°C, and the highest value was 2217 µS cm^{-1} measured in the chicken biochar produced at 700°C. The pH values for the ten biochars in this experiment ranged from 8.0 to 10.3. These biochar N, K, EC, and pH values are relatively similar to those reported by other investigators (Rajkovich et al., 2012; Revell et al., 2012; Tsai et al., 2012; Uzoma et al., 2011; Wang et al., 2012). The mean quantities of N and K added to each pot along with the biochar were 153 ±108 and 194 ±98 mg, respectively.

The ash content of the biochars varied from 24.2% to 52.9%. It was lowest in the dairy biochar processed at 350°C and highest in the swine biochar processed at 700°C. In other investigations, ash contents of poultry manure-based biochars have ranged from 47% to 57% (Rajkovich et al., 2012; Revell et al., 2012). For dairy manure-based biochars, ash contents have been reported to range from 14% to 38% (Rajkovich et al., 2012; Wang et al., 2012). In the case of swine based-biochars, the reported range of ash contents has been 43% to 53% (Tsai et al., 2012).

The ratios of the secondary plant nutrients (Ca and Mg) and the micronutrients (Zn and Cu) relative to the total P of the biochars are presented in table 3. In all cases, the Ca content was equal to or larger than the P content. Its lowest ratio was 1.01 for the swine 350°C biochar, and its highest ratio was 2.67 for the dairy 350°C biochar. The lowest ratio of Mg was 0.32 for the turkey 350°C biochar, and the highest was 1.22 for the dairy biochar with either pyrolysis temperature. The dairy biochar had distinctly higher relative contents of Ca and Mg than did any of the other biochars. While the ratios of Zn and Cu were about a thousand times lower, their additions were important because they are both necessary for plant growth and potentially toxic to plant growth. In the case of both elements, the ratios were much higher in swine biochar than in the other biochars. While these levels are sufficiently high to be noted relative to very long-term application to the soil, they were neither significantly different from levels applied in standard manure applications nor sufficiently high to be of concern for application at rates required for crop P needs.

Table 3. Ratio of secondary and micro plant nutrients relative to total P of the manure biochars.

Feedstock	Pyrolysis Temp. (°C)	Ca (g g^{-1} P)	Mg (g g^{-1} P)	Zn (mg g^{-1} P)	Cu (mg g^{-1} P)
Dairy	350	2.67	1.22	36.10	9.90
	700	2.65	1.22	25.03	9.64
Beef	350	1.99	0.67	31.49	8.04
	700	1.99	0.69	25.45	7.73
Chicken	350	1.28	0.45	34.23	10.24
	700	1.29	0.46	32.37	9.94
Turkey	350	1.54	0.32	26.34	20.42
	700	1.53	0.34	24.84	24.84
Swine	350	1.01	0.63	81.77	39.54
	700	1.04	0.63	84.42	41.46

SOIL NUTRIENTS

When the chemical fertilizer P was added to the soil of this experiment, it produced the desired linear increase of Mehlich 3 P (tables 1 and 4). The linear response curve was as follows: Mehlich P (mg g^{-1}) = 0.28 (applied P fertilizer) + 10.0; the R^2 value for this equation was 0.98. The soil Mehlich extractable P values for the P-0, P-50, and P-150 fertilizer treatments were 10.6, 23.3, and 51.5 µg P g^{-1} soil, respectively (table 4). These values were all significantly different from each other ($p \leq 0.05$). When the manure biochars were added at the rate of 50 mg P_2O_5 kg^{-1} soil, the Mehlich P had a mean of 21.5 ±1.9 µg P g^{-1} soil. This was very close to the value of 23.3 µg P g^{-1} soil for the P-50 treatment. The soil M3 P was not significantly different based on processing temperatures. The dairy biochar soil had the highest mean value of 24.4 µg P g^{-1} soil. While this

Table 4. Impact of manure biochars and chemical fertilizer on soil nutrient concentrations.[a]

Amendment		N (g kg^{-1})	C (g kg^{-1})	P (µg g^{-1} soil)	K (µg g^{-1} soil)	Ca (µg g^{-1} soil)	Mg (µg g^{-1} soil)	Zn (µg g^{-1} soil)	Cu (µg g^{-1} soil)
350°C biochar[b]	Chicken	1.00 a-d	28.8 abc	21.8 cd	45.9 d-g	807 b-d	34.6 cde	4.0 c-f	0.42 abc
	Dairy	1.05 abc	28.1 a-d	25.4 b	41.0 fgh	840 a-d	46.4 a	5.4 abc	0.32 bc
	Beef	0.97 b-e	27.7 b-e	23.2 bc	52.0 cde	825 a-d	38.0 bc	5.2 a-d	0.34 abc
	Turkey	1.00 a-d	28.2 a-d	20.5 d	44.4 e-h	829 a-d	30.7 f	3.7 d-h	0.35 abc
	Swine	1.03 a-d	29.6 a	19.6 d	40.5 fgh	798 bcd	32.6 def	2.8 f-i	0.47 ab
	Mean	1.01 ±0.03	28.5 ±0.7	22.1 ±2.3	44.8 ±4.6	820 ±17	36.5 ±6.2	4.2 ±1.1	0.38 ±0.06
700°C biochar	Chicken	0.95 cde	26.0 ef	19.9 d	48.3 c-f	865 ab	34.9 cd	6.7 a	0.41 abc
	Dairy	1.04 abc	29.0 ab	23.4 bc	47.6 c-f	878 a	39.3 b	4.0 c-f	0.40 abc
	Beef	0.89 e	25.6 f	20.7 d	54.5 bc	855 abc	41.1 b	4.5 b-e	0.36 abc
	Turkey	0.97 b-e	28.3 a-d	19.7 d	39.7 gh	856 ab	31.3 ef	2.2 hi	0.50 a
	Swine	0.96 cde	28.0 a-d	20.7 d	37.0 h	823 a-d	33.0 def	3.8 c-g	0.42 abc
	Mean	0.96 ±0.05	27.4 ±1.5	20.9 ±1.5	45.4 ±7.1	856 ±20	35.9 ±4.2	4.2 ±1.6	0.42 ±0.05
Grand mean		0.99 ±0.05	27.9 ±1.3	21.5 ±1.9	45.1 ±5.6	838 ±26	36.2 ±5.0	4.2 ±1.3	0.40 ±0.06
Fertilizer	P-150	1.05 abc	27.1 c-f	51.5 a	61.0 ab	871 ab	25.3 g	2.4 ghi	0.29 c
	P-50	1.07 a	27.2 b-f	23.3 bc	52.8 cd	870 ab	26.8 g	2.7 f-i	0.41 abc
	P-0	0.93 de	26.6 def	10.6 e	67.3 a	774 d	26.0 g	1.9 i	0.32 bc
	N only	0.96 cde	28.2 a-d	10.9 e	37.3 h	879 a	27.1 g	3.2 e-i	0.30 c
	Blank	1.06 ab	27.3 b-f	10.3 e	36.7 h	781 d	27.2 g	6.0 ab	0.37 abc

[a] Means followed by the same letter are not significantly different at the 0.05 level by the least means square procedure.

[b] Biochar was added to apply 50 mg P kg^{-1} soil.

soil was initially low in P, it needed additional P to be in the range of a normal agronomic field. Accordingly, with the addition of either the fertilizer P-50 treatment or the biochars, the soil P was increased to a level somewhat similar to the control treatment soil in the poultry biochar study by Revell et al. (2012). These values would not be considered high for soil test values. However, they were more than 10 times lower than the soil P obtained when 5% biochar was added to the Revell soils. Thus, the biochars of the current study were in the desired range for evaluation of their application as fertilizer material, as opposed to C sequestration amendments with P pollution potential.

The P fertilizer treatments had an addition of K; consequently, they were significantly higher in soil K content than the blank or N-only treatments. The blank soil treatment yielded 3.7 μg K g^{-1} soil, while the fertilizer treatments ranged from 52.8 to 67.3 g K g^{-1} soil. The biochar treatments had a mean of 45.1 ±5.6 μg K g^{-1} soil. The soil K concentrations of the biochar treatments with 350°C and 700°C biochars varied by less than 1 mg. The biochar K mean was significantly lower than the fertilizer treatment mean of 60.4 μg g^{-1} soil. Neither treatment would have been considered to have high soil K (Heckman and Kamprath, 1992). As with K and P, the soil was initially low in N. The soil of the blank treatment had 1.1 g N kg^{-1} soil. This was only 25% of the soil N content of an Autryville loamy sand used for swine wastewater irrigation and bermudagrass growth (Stone et al., 2008). The addition of 50 mg N kg^{-1} soil to all treatments other than the blank provided sufficient N for ryegrass growth. The mean soil N content for the biochar treatment was 0.99 ±0.05 g N kg^{-1} soil. The soil N was not significantly different based on the temperature of the biochar ($p \leq 0.5$), and the range was <0.2 g N kg^{-1} soil. The blank treatment had a soil C content of 27.3 g C kg^{-1} soil. This soil C content was somewhat high for a sandy soil compared to those even under conservation or standard wastewater spray fields (Hunt et al., 1996, 2011) and was the result of the soil being recently under timber. Thus, the small addition of biochar for fertilizer increased the soil C very little. The biochar soil treatment mean was 27.9 ±1.3 g C g^{-1} soil, and the range was 25.6 to 29.6 g C kg^{-1} soil. Thus, the biochar soils had a high C:N ratio of 28.

As a result of the liming, the Ca content of the soil was relatively similar (table 4). The mean for the fertilizer treatments was 870 ±1 μg Ca g^{-1} soil. This was comparable to the biochar treatments, which had a mean of 838 ±26 μg Ca g^{-1} soil. In contrast, the biochar treatment Mg mean of 36.2 ±5.0 μg g^{-1} soil was higher than the fertilizer treatment mean of 26.0 μg g^{-1} soil.

With regard to Zn and Cu, the fertilizer treatments had a Zn mean of 2.6 ±0.54 μg g^{-1} soil and a Cu mean of 0.33 ±0.05 μg g^{-1} soil. While there were variations in the Zn and Cu soil contents of the fertilizer and manure biochar treatments, none of the treatments were at a level reported to be a likely problem for plant growth, particularly at near-neutral soil pH levels (Fan et al., 2011; He et al., 2006; Novak et al., 2004). The highest Zn concentration was 6.7 μg Zn g^{-1} soil in the chicken 700°C biochar treatment. The highest Cu was 0.50 μg Cu g^{-1} soil in the turkey 700°C treatment.

RYEGRASS DRY MATTER YIELD

The ryegrass dry matter accumulation (yield) was not significantly affected by the biochar processing temperature ($p \leq 0.05$). However, the poultry biochar produced higher yields than the other feedstocks, and the swine biochar produced lower yields than any other feedstock ($p \leq 0.05$). As anticipated, the blank treatment produced both the lowest ryegrass harvest height of 11.4 cm and lowest dry matter accumulation of 2.74 g (table 5). The highest height was

Table 5. Impact of biochar and chemical fertilization on ryegrass harvest yields, biomass nutrient concentrations.[a]

Amendment	Plant Height (cm)	Dry Matter (g)	N (mg g^{-1})	P (mg g^{-1})	K (mg g^{-1})	Ca (mg g^{-1})	Mg (mg g^{-1})	Zn (mg g^{-1})	Cu (mg g^{-1})	HHV (MJ kg^{-1})
350°C biochar[b]										
Chicken	17.63 a	9.23 a	28.7 d-g	3.5 cde	24.5 b	9.2 h	2.3 efg	0.056 cde	0.014 ab	17.65 a-c
Dairy	17.23 ab	7.80 b	28.3 efg	4.4 b	20.3 cd	11.1 ef	2.8 bc	0.061 b-e	0.016 ab	17.55 a-d
Beef	15.92 bc	7.54 b-d	27.4 fg	4.0 bc	28.4 a	9.1 h	2.2 efg	0.100 a	0.015 ab	17.37 b-d
Turkey	15.92 bc	7.47 b-d	29.5 c-f	3.1 d-h	22.1 c	11.5 de	2.3 def	0.051 de	0.015 ab	17.79 a
Swine	15.84 bc	6.63 d	30.8 cd	3.5 c-f	14.6 g	13.9 ab	3.2 a	0.052 de	0.013 ab	17.51 a-d
Mean	16.51 ±0.85	7.73 ±0.94	28.9 ±1.3	3.7 ±0.5	22.0 ±5.1	11.0 ±2.0	2.6 ±0.4	0.064 ±0.021	0.015 ±0.001	17.57 ±0.16
700°C biochar										
Chicken	16.49 abc	8.11 b	26.9 gh	2.9 e-h	25.0 b	9.9 fgh	2.0 gh	0.053 cde	0.016 ab	17.24 d
Dairy	17.15 ab	7.96 b	29.1 c-g	2.8 g-h	18.8 de	10.7 efg	2.6 cd	0.062 b-e	0.020 a	17.45 a-d
Beef	16.27 abc	7.81 b	28.0 fg	2.9 e-h	27.7 a	11.0 ef	2.0 fgh	0.059 b-e	0.014 ab	17.21 d
Turkey	16.47 abc	7.95 b	28.7 d-g	2.9 fgh	21.8 c	11.1 ef	2.3 de	0.050 e	0.013 ab	17.36 b-d
Swine	16.01 abc	6.84 cd	31.1 c	3.4 d-g	15.7 fg	14.5 a	3.2 a	0.054 cde	0.017 ab	17.71 a-c
Mean	16.48 ±0.42	7.73 ±0.51	28.8 ±1.6	3.0 ±0.3	21.8 ±4.8	11.4 ±1.8	2.4 ±0.5	0.056 ±0.005	0.016 ±0.003	17.40 ±0.20
Grand mean	16.49 ±0.64	7.73 ±0.72	28.9 ±1.3	3.3 ±0.5	21.9 ±4.7	11.2 ±1.8	2.5 ±0.4	0.060 ±0.015	0.015 ±0.002	17.48 ±0.19
Fertilizer										
P-150	16.92 ab	7.61 bc	28.8 d-g	6.2 a	25.3 b	13.2 abc	2.3 def	0.059 b-e	0.011 b	17.56 a-d
P-50	16.83 ab	7.24 b-d	30.3 cde	3.6 cd	24.7 b	12.6 cd	2.2 efg	0.064 bcd	0.014 ab	17.35 cd
P-0	14.99 cd	5.63 e	35.1 b	1.3 i	29.1 a	10.3 e-h	1.9 h	0.065 bc	0.014 ab	17.34 cd
N only	13.79 d	5.14 e	40.3 a	1.5 i	17.0 ef	12.9 bc	3.1 ab	0.070 b	0.012 ab	17.74 ab
Blank	11.43 e	2.74 f	25.1 h	2.7 h	22.0 c	9.4 gh	2.2 e-h	0.059 b-e	0.019 a	NA[c]

[a] Means followed by the same letter are not significantly different at the 0.05 level by the least means square procedure.
[b] Biochar was added to apply 50 mg P kg^{-1} soil.
[c] NA = not enough material was available for analysis.

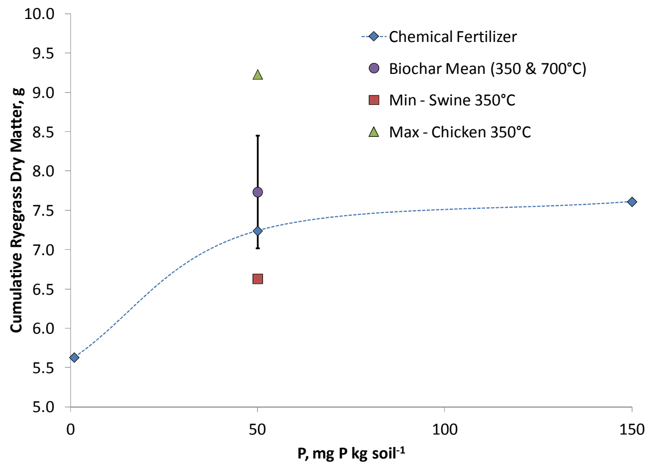

Figure 1. Ryegrass growth response curve to chemical P fertilization and yield response to P added via livestock on a P_2O_5 basis.

17.6 cm for the chicken 350°C treatment. The plant heights were well correlated with the accumulated dry matter: plant height (cm) = 0.95 (dry weight) − 8.12; the R^2 value was 0.91. The ryegrass dry matter accumulation (yield) and soil P concentration are shown in figure 1. The yield was 5.63 g at the P-0 level and 7.61 g at the P-150 level. The fertilizer P-50 level treatment had a yield of 7.24 g. With the exception of the swine biochar treatments, all of the biochars produced yields higher than the P-50 treatment. The swine 350°C biochar yield of 6.63 g was the lowest, and the chicken 350°C yield of 9.23 g was the highest. The least amount of biochar, 0.03%, was added in the swine 700°C biochar treatment; the greatest amount of biochar, 0.19%, was added in the dairy 350°C treatment. These values are small relative to the typical addition of biochar. For instance, Revell et al. (2012) added 0.5% to 5% in their investigation of poultry biochar and peppers, and Rajkovich et al. (2012) added 0.2% to 7% biochar in their investigation, which used dairy and poultry biochars. Thus, they were also adding the accompanying higher levels of P to the soil. There were also accompanying addition of micronutrients, such as Zn and Cu, and these have the potential of either increasing or decreasing yield. However, in the present study, none of these accompanying nutrient additions were well correlated to dry matter yield.

Nutrient Concentrations and Energy Content

With regard to the P concentration of the ryegrass, the biochars were different relative to processing temperature (table 5). The 700°C biochar had a mean of 3.0 mg P g^{-1}, and the 350°C biochar had a mean of 3.7 mg P g^{-1}. The mean for all of the biochars was 3.3 ±0.5 mg P g^{-1}. This mean was very close to the 3.6 mg P g^{-1} of the P-50 treatment. It was much higher than the 2.7 mg g^{-1} of the blank treatment. The minimum and maximum P concentrations for the biochar-treated ryegrass were 2.8 and 4.4 mg g^{-1}. However, none of the treatments were close to the concentration of 6.2 mg P g^{-1} in the P-150 treated ryegrass. This result documents the accumulation of P in excess of the plant growth requirement.

For the other measured nutrients associated with biochar amendment (table 5), all of their concentrations were in the range of those reported as typical by Whitehead (2000). The mean ryegrass concentration of N was 28.9 ±1.3 mg g^{-1}. The means for K, Ca, and Mg were 21.9 ±4.7, 11.2 ±1.8, and 2.5 ±0.4 mg g^{-1}, respectively. The Zn and Cu ryegrass concentrations were 0.060 ±0.015 and 0.015 ±0.002 mg g^{-1}, respectively. However, the Zn and Cu concentrations were on the high side of Whitehead's typical concentrations. This is important because Zn and Cu accumulation can cause plant growth and toxicity problems (Paramasivam et al., 2009; Zhang et al., 1997). While these concentrations serve as a caution against prolonged application of manure biochars to very sandy soils such as the Uchee, they are not even close to the concentrations reported for polluted soils, such as the 30 mg Cu kg^{-1} in ryegrass shoots grown on contaminated soil by Karami et al. (2011). Concerning processing temperatures and feedstocks, there were no significant differences in ryegrass nutrient concentrations between the 350°C and 700°C biochars. Relative to the feedstocks, the swine biochar produced ryegrass with higher concentration of N, Ca, and Mg than any of the other biochars ($p \leq 0.05$) and the lowest K. However, the Zn and Cu ryegrass concentrations were not significantly different based on biochar feedstock ($p \leq 0.05$).

When compared to the chemical fertilized treatment, the nutrient concentrations of the biochar-treated ryegrass were generally slightly higher than the blank and similar to the fertilizer treatments. For P, the maximum and minimum concentrations of the biochar treatments were 4.4 and 2.8 mg g^{-1} for the dairy 350°C and 700°C biochars, respectively. The blank treatment was lower, and the P-50 treatment was within this range of N concentrations. Of course, the P-150 treatment resulted in a higher P concentration of 6.2 mg g^{-1} in the ryegrass, even though it did not induce a higher yield. The N concentrations of the P-50 and blank treatments were similar to the P concentrations in their relation to the biochar treatment. In the case of K, Ca, Mg, Zn, and Cu, the P-50 and blank treatments were within the minimum-maximum range of the biochar ryegrass concentrations.

With regard to HHV, no single biochar treatment produced ryegrass with a statistically different HHV. The turkey 350°C biochar treatment produced ryegrass with the greatest energy content of 17.79 MJ kg^{-1} (table 5), and the lowest HHV was associated with the beef 700°C biochar. Biochar temperature affected the HHV of ryegrass grown with chicken and turkey biochars; lower-temperature treatments generated ryegrass biomass with greater HHV. Averaged across all biochar treatments (17.48 MJ kg^{-1}; table 5), biochar treatments were found to have a significant contrast with the N-only treatment ($p < 0.08$), which exhibited one of the greater HHV of 17.74 MJ kg^{-1}. All of the HHV presented were lower than that of bermudagrass grown during an outdoor irrigation experiment (Cantrell et al., 2009; Stone et al., 2008); irrigating bermudagrass with a commercial fertilizer (270 kg N ha^{-1}) gave HHV of 18.9 to 19.1 MJ kg^{-1}, while irrigating bermudagrass with treated swine effluent (to provide 270 kg N ha^{-1}) yielded HHV ranging from 18.7 to 18.9 MJ kg^{-1}.

Nutrient Accumulation by Ryegrass

The ryegrass P accumulation of the 350°C biochar treatments was 30.6 mg (table 6). This was significantly higher ($p \leq 0.05$) than the 24.6 mg P in the 700°C biochar treatments. The manure biochars were not significantly different in their ryegrass P accumulations ($p \leq 0.05$). The mean for all of the biochars was 27.9 ±4.8 mg P. The maximum accumulation for the biochar treatments was 36.1 mg in the dairy 350°C biochar, and the minimum accumulation was 23.6 mg P in the dairy 700°C biochar. This minimum accumulation was not significantly lower than the P-50 treatment; however, it was dramatically greater than the blank, N-only, and P-0 treatments. These treatments accumulated <8 mg P. Following the high concentration of P in the ryegrass of the P-150 treatment, it accumulated the maximum quantity of P (49.3 mg).

With regard to ryegrass N accumulation, the processing temperature caused no significant difference (table 6). The mean N accumulation was 220 ±16 mg. The maximum accumulation of N for the biochar treatments was 262 g in the chicken 350°C biochar. This accumulation was greater than any other treatment. The minimum accumulation was 204 g, which was accumulated by the ryegrass grown on the swine 350°C biochar. It was not significantly different ($p \leq 0.05$) from the P-0, P-50, or N-only treatments. However, it was dramatically different from the 67 mg of N accumulated in the blank treatment.

For the other nutrients (K, Ca, Mg, Zn, and Cu), only Zn accumulation was significantly affected by processing temperature (table 6). Zn was also the only nutrient whose ryegrass accumulation was affected by feedstock; the Zn accumulation in the beef manure treatment was higher than in any other treatment. The mean K accumulation for the biochars was 168.6 mg, which was similar to the P-50 treatment but much higher than the blank. The same treatment relationship was true for Ca, Mg, Zn, and Cu with the following means and standard deviations: 86.0 ±7.7, 19.8 ±2.5, 0.47 ±0.13, and 0.13 ±0.03 mg, respectively. These quantities removed by the plant constituted <1% of the quantity applied with the biochar.

Conclusions

Phosphorus as measured by Mehlich 3 extraction was readily available from all the manure-derived biochars. These biochars did not contain yield-limiting levels of EC, pH, Zn, or Cu. Moreover, the biochar P was readily taken up by ryegrass plants. The biochar treatments generally produced ryegrass dry matter yields similar to or greater than chemical fertilized ryegrass. The chicken 350°C biochar generated the largest ryegrass yield, and the swine 350°C biochar produced the lowest yield. For the biochar treatments, the ryegrass Cu and Zn concentrations were within normal ranges, and they were not significantly different from the chemical fertilization treatments. The manure biochars also have the inferred advantage of temperature destruction of pathogens. Additionally, their increased density allows the possibility of greater shipping distances. These manure-based biochars present a new and interesting way to manage manures to optimize the use of P for production of grasses such as annual ryegrass. Furthermore, these manure-based biochars present a new approach to reducing the demand on finite P global reserves.

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Table 6. Impact of manure biochars and chemical fertilizer on ryegrass biomass nutrient accumulation.[a]

Amendment		N (mg)	P (mg)	K (mg)	Ca (mg)	Mg (mg)	Zn (mg)	Cu (mg)
350°C biochar[b]	Chicken	262 a	34.5 bc	222.4 a	87.2 a-c	22.0 a-b	0.53 b	0.15 a-c
	Dairy	219 b	36.1 b	157.0 e-f	87.0 a-c	22.5 a	0.48 b-c	0.14 a-c
	Beef	208 b-c	31.6 b-d	213.1 a-b	68.7 d-e	17.3 d	0.78 a	0.12 a-c
	Turkey	215 b-c	26.8 de	162.1 e-f	85.5 b-c	18.7 c-d	0.39 c-f	0.14 a-c
	Swine	204 b-c	24.2 e	97.5 g	92.6 a-c	22.1 a-b	0.34 f	0.09 b-c
	Mean	222 ±23	30.6 ±5.0	170.4 ±50.2	84.2 ±9.1	20.5 ±2.4	0.50 ±0.17	0.13 ±0.02
700°C biochar	Chicken	215 b-c	24.9 de	201.8 a-c	80.4 c-d	16.3 d	0.42 c-f	0.15 a-b
	Dairy	226 b	23.6 e	144.5 f	85.9 a-c	21.2 a-c	0.48 b-d	0.18 a
	Beef	218 b	24.5 e	210.0 a-b	87.5 a-c	16.5 d	0.48 b-c	0.12 a-c
	Turkey	224 b	23.9 e	169.1 d-f	87.0 a-c	19.1 b-d	0.39 c-f	0.11 a-c
	Swine	208 b-c	25.0 de	108.6 g	98.2 a-b	22.5 a	0.37 d-f	0.14 a-c
	Mean	218 ±7	24.6 ±0.5	166.8 ±41.8	87.8 ±6.5	19.1 ±2.8	0.43 ±0.05	0.14 ±0.03
Grand mean		220 ±16	27.9 ±4.8	168.6 ±43.6	86.0 ±7.7	19.8 ±2.5	0.47 ±0.13	0.13 ±0.03
Fertilizer	P-150	216 b-c	49.3 a	194.0 b-d	100.3 a	18.0 c-d	0.45 b-e	0.09 b-c
	P-50	216 b-c	28.1 c-e	179.6 c-e	91.4 a-c	16.9 d	0.48 b-d	0.11 a-c
	P-0	191 c	7.9 f	165.6 e-f	57.0 e	10.5 e	0.37 e-f	0.09 b-c
	N only	201 b-c	7.9 f	85.1 g	64.5 e	16.7 d	0.37 d-f	0.07 b-c
	Blank	67 d	7.8 f	59.8 h	25.2 f	6.4 f	0.16 g	0.07 c

[a] Means followed by the same letter are not significantly different at the 0.05 level by the least means square procedure.

[b] Biochar was added to apply 50 mg P kg^{-1} soil.

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